***JPL* ANALYTICAL CHEMISTRY LABORATORY**

*Analytical Chemistry and Materials Development Group 3531 ACL-VLG-042221-1*

### To: Calum Torrie, Chandra Romel, Matt Heintze 04-22-21

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**Subject:** LIGO-E 2100134-v4; FTIR HAM 7

**Background**

Several hexane swabs were used to sample System inside HAM 7 at various locations. Bake Load: Post Bake, Class A. The samples plus a control swab were submitted to the ACL for analysis to determine the level molecular cleanliness. Cleanliness requirement for surfaces after an air bake is level A/50 or ≤ 0.02 micrograms/cm2 and ≤ 0.4 microgram/hole for through holes and ≤ 0.7 microgram/hole for blind holes (9).

**Results**

* The molecular analysis results performed by FTIR can be seen in **Table 1**. Total amounts were reported as micrograms per centimeter squared or micrograms per hole.
* All “Surface” samples fail to meet level R2E-2 or A/50 of IEST-STD-CC1246E.
* “hole” sample (sample #7) failed to meet the requirement of ≤ 0.4 microgram/hole and ≤ 0.7 microgram/hole; the type of hole was not specified.

 **Table 1: FTIR Results for the Hexane Swab Samples**

|  |  |  |  |
| --- | --- | --- | --- |
| PARTS |  | SAMPLES | **RESULTS** |
| # | Part No. | SN | Description | PO # | Vial # | Type | Description | Amount  | **Chemical Functional Group** | **Total Amount** | **Pass/Fail** |
| 1 | S1 in Kit 1 |  | Before on floor | S491485-A+ | 1 | Surface | Chamber floor center north side | Area: 154.8 cm2 | AHC, Ester | 0.60 μg/cm2 | **Fail** |
| Holes | - | # of Holes: - | - | - | **-** |
| 2 | S2 in Kit 1 |  | After cleaning on floor | S491485-A+ | 2 | Surface | Same as S1 | Area: 154.8 cm2 | AHC, Ester | 1.4 μg/cm2 | **Fail** |
| Holes | - | # of Holes: - | - | - | **-** |
| 3 | S3 in Kit 2 |  | Before middle of north support tube | S491485-A+ | 3 | Surface | Support tube north between holes 4 &5 east side | Area: 103.2cm2 | AHC, Ester |  0.16 μg/cm2 | **Fail** |
| Holes | - | # of Holes: - | - | - | **-** |
| 4 | S4 in Kit 2 |  | After cleaning on north support tube | S491485-A+ | 4 | Surface | Same as S3 | Area: 103.2 cm2 | AHC, Ester | 1.0 μg/cm2 | **Fail** |
| Holes | - | # of Holes: - | - | - | **-** |
| 7 | S7 in Kit 3 |  | All support tube holes in south support tube | S491485-A+ | 4 | Surface | - | Area: - cm2 | - | - | **-** |
| Holes | Center 7 holes in south support tube | # of Holes:  | AHC | 17.8 μg/hole | **Fail** |

**Terminology:**

AHC: Aliphatic Hydrocarbons, base oil of lubricants, additives

Ester: Common sources include plasticizers (such as DOP) and finger prints

μg/cm2: micrograms per square centimeter

“-“ Not applicable/ no sample submitted

**Experimental**

A hexane (NVR) swab (Soxhlet extracted for 48hrs) was used to extract low volatile residues (LVR) on a surface. LVR dissolved into the hexane were analyzed using Diffuse Reflectance Infrared Fourier Transform (DRIFT) spectroscopy. FTIR provides chemical functional group information for qualitative and quantitative determination of materials. The analysis complies with IEST-STD-CC1246E and M2020 requirements (1-2), using a published methodology (3-7), and is sensitive to the stringent levels of molecular contamination (8).

**DRIFT- FTIR References and Notes**

1. The method conforms to the Institute of Environmental Science and Technology (IEST), Contamination Control Division Document IEST 1246E “Product Cleanliness Levels and Contamination Control Program”. The method is intended to conservatively bin the molecular contamination into cleanliness levels. The ACA M2020 limit is “Level R1E-1” (level A/10 of IEST-STD-CC1246E) and this is 0.1 microgram per square centimeter (μg/cm2) and this corresponds to an average film thickness of 10 angstroms (assuming a density of 1.0).
2. M. S. Anderson, “The Chemical Analysis Plan for M2020 Sample Return Hardware”, IOM-3530-2018-043.
3. Fuller, Michael P., and Peter R. Griffiths. "Infrared micro-sampling by diffuse reflectance Fourier transform spectrometry." Applied Spectroscopy 34, no. 5 (1980): 533-539.
4. “Diffuse Reflection spectroscopy” Handbook of Vibrational Spectroscopy, Volume 2, page 1125-1175, J. C. Chalmers and P. R. Griffiths, eds., John Wiley & Sons, Chichester, UK, pp. 2263 (2002).
5. Averett, Lacey A., and Peter R. Griffiths. "Method to improve linearity of diffuse reflection mid-infrared spectroscopy." Analytical chemistry 78, no. 23 (2006): 8165-8167.
6. M. S. Anderson et al "Analysis of Semi-Volatile Residues Using Diffuse Reflectance Infrared Fourier Transform Spectroscopy" in Optical System Contamination: Effects, Measurements, and Control VII; July 2002, edited by Phillip T. C. Chen and O. Manuel Lee; Proceedings of the SPIE, Vol. 4774, pp. 251-261, (2002).
7. Handbook of Vibrational Spectroscopy, Volume 3, J. C. Chalmers and P. R. Griffiths, eds., John Wiley & Sons, Chichester, UK, pp. 2263 (2002).
8. Very clean surfaces, ≤0.02 μg/cm2, with mono-molecular layers or less are more complex to describe when cleaning or analyzing. Carbon/hydrocarbon based substances are known to rapidly (within ~1 hour) accumulate on most, if not all, freshly exposed surfaces. This “adventitious” carbon is well documented in clean rooms and vacuum systems and compositionally varies by environment. Adventitious carbon is a discontinuous layer of approximately ~0.2 nanometers thick or ~0.02 µg/cm2 up to 0.1 µg/cm2 (for ρ = 1). The last mono-layer fractions may in some cases be strongly adsorbed to the surface as a “corrosion” layer. Therefore solvent based sampling methods may not remove these fractions, particularly if the surface is porous. When specifying cleanliness level to lower than A/10 IEST-STD-CC1246E (0.1 µg/cm2) these monolayer effects become more significant. See Anderson M. S., “Chemical Analysis and Mitigation of Adventitious Carbon Contamination”, 1/3/2017, Mars 2020 Project, JPL D-97858. See also: H. Piao and N. S. McIntyre, “Adventitious carbon growth on aluminum and gold–aluminum alloy surfaces”, *Surface and Interface Analysis*, 2002; 33: 591–594.
9. Torrie, C., Coyne, D. “FTIR Testing to Qualify Parts for LIGO UHV Service,” Doc No. E0900480- Rev. v5., Oct. 2017.

<https://dcc.ligo.org/public/0007/E0900480/005/E0900480-v5%20FTIR%20testing.pdf>